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URBANA

REPORT OF INVESTIGATIONS—NO. 72

PETROGRAPHIC AND CERAMIC PROPERTIES OF PENNSYLVANIAN SHALES OF ILLINOIS

BY

RALPH E. GRIM

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Topographic Mapping in Cooperation with the United States Geological Survey.

This Report is a Contribution of the Division of Petrography.

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PETROGRAPHIC AND CERAMIC PROPERTIES OF PENNSYLVANIAN SHALES OF ILLINOIS*

By Ralph E. Grim

ABSTRACT

"Coal Measures" shales, which are widespread in Illinois, are used extensively as a ceramic raw material. Ceramic determinations are given for a large number of shales to illustrate the wide range in their properties. The mineral composition, base-exchange capacity, and textural characteristics are presented for the same shales, and the relation of these characteristics to the ceramic properties of the shales is analyzed.

INTRODUCTION

The bedrock formations immediately underlying about three fourths of the State of Illinois are Pennsylvanian or "Coal Measures" in age and are made up of a large number of beds of sandstone, underclay, coal, limestone, and shale. Many different beds of Pennsylvanian shale as a consequence are to be found cropping out in a large part of the State. These shales are extensively used as ceramic raw material.

The purpose of this research was to investigate the range of ceramic properties of the Pennsylvanian shales; to determine their mineral composition, base-exchange capacity, and texture; and to study some relationships between these factors of composition and ceramic properties. This work is part of the study of the Illinois State Geological Survey of the fundamental factors determining the properties of clays and shales found in Illinois.

PROCEDURE

Samples were collected (table 1) from the working pits of many ceramic plants to obtain examples of shales with a wide range of composition and ceramic properties. Where the characteristics of the shale varied in any pit, samples were collected from individual zones in which the characteristics were constant.

Complete mineralogical analyses, based on microscopic, X-ray, and chemical data, were made for each sample. Textural characteristics were obtained by sedimentation analyses and by the study of thin sections with the petrographic microscope. Ceramic tests of each sample were made by the Department of Ceramic Engineering of the University of Illinois.

CERAMIC PROPERTIES

The ceramic properties (table 2) of the Pennsylvanian shales show wide variations. The water plasticity, for example, varies from 38.5 to 21.0 per cent, the modulus of rupture from 1076 to 162 pounds per square inch, the volume drying shrinkage from 35.6 to 13.0 per cent, and the volume firing shrinkage from 33.3 to 18.3 per cent. Except for two samples, No. 254 and No. 268, which were gray, the fired color is uniformly red. The shales are not refractory (P.C.E. 3 to 15) and fire at relatively low temperatures (cones 08 to 02).

TEXTURE

To study the relation between mineral composition, texture, and ceramic properties of these shales, the amount of clay minerals, whatever their particle size, and grains of all other minerals smaller than 0.002 mm. were determined microscopically for each shale by running many traverses across several thin sections of each shale. Values for the total amount of clay minerals and grains of other minerals smaller than 0.002 mm., designated for convenience as fraction A, are given in column (2) of Table 2.

In figures 1 to 6, certain ceramic properties are plotted against the amount of fraction A. These figures show that the total amount of fraction A is an exceedingly important factor in controlling the ceramic properties of these shales. Figures 1, 2, and 3 show that the water of plasticity, modulus of rupture, and drying shrinkage increase

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*Presented at the Forty-First Annual Meeting, The American Ceramic Society, Chicago, Ill., April 18, 1339 (Materials and Equipment Division).

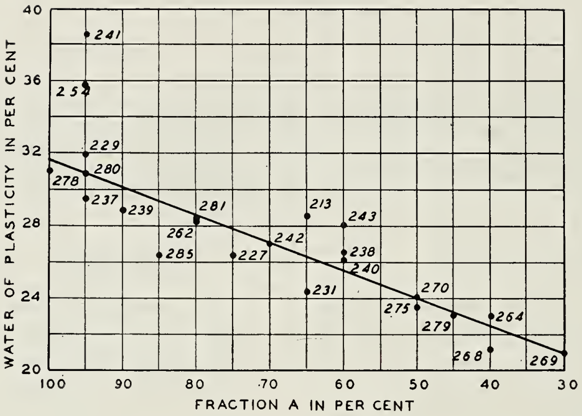

Fig. 1.—Water of plasticity plotted against amount of fraction A.

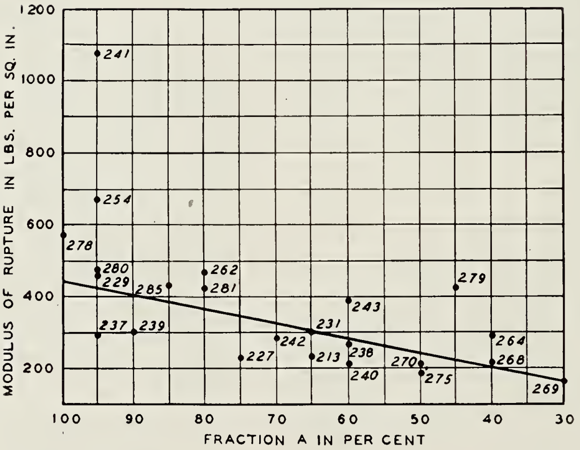

Fig. 2.—Modulus of rupture plotted against amount of fraction A.

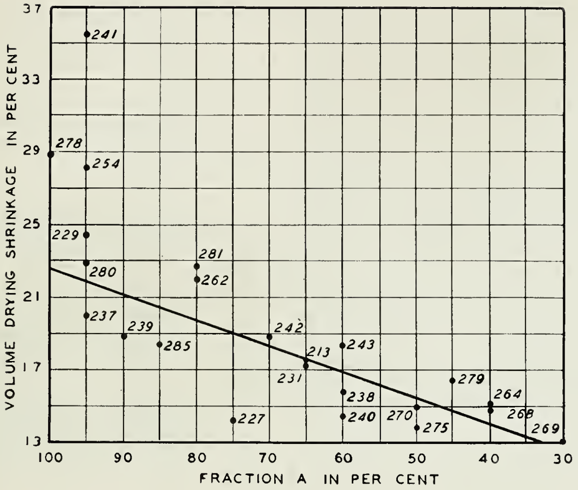

Fig. 3.—Volume drying shrinkage plotted against amount of fraction A.

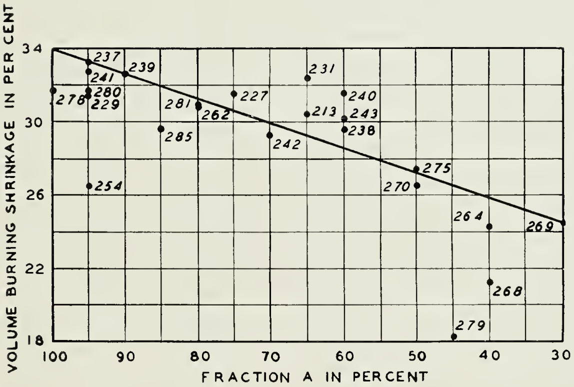

Fig. 4.—Volume burning shrinkage plotted against amount of fraction A.

Table 1.—Samples Investigated

Sample No.	Location	Stratigraphic Position
278	Albion Brick Co., Albion Plant No. 2; lowermost 6 ft.	Upper Pennsylvanian shale
280	Hydraulic Press Brick, Edgemont; upper shale	Shale overlying Collinsville limestone
229	Carlinville Tile Co., Carlinville; entire shale	Shale overlying Shoal Creek limestone
241	White Hall Sewerpipe & Stoneware Co., White Hall; uppermost shale	Upper part of Purington shale
254	Sheffield Clay Products Co., Sheffield; lower 8 ft.	Sheffield shale overlying coal No. 6
237	Springfield Clay Products Co., Petersburg; lower 12 ft.	Farmington shale overlying coal No. 7
239	Springfield Clay Products Co., Petersburg; upper 15 ft.	" " " " " "
285	Western Brick Co., Danville; entire shale	" " " " " "
262	Albion Brick Co., Albion Plant No. 1; lower 18 ft.	Upper Pennsylvanian shale
281	Richards Brick Co., Edwardsville; lower 6 ft.	Shale above Trivoli limestone
227	Hydraulic Press Brick Co., Sparland; entire shale	Farmington shale overlying coal No. 7
242	White Hall Sewerpipe & Stoneware Co., White Hall; lower shale	Purington shale overlying coal No. 2
231	Sheffield Clay Products Co., Sheffield; upper 12 ft.	Sheffield shale overlying coal No. 6
213	Hydraulic Press Brick Co., Shale City; entire shale	Gilchrist shale under coal No. 2
243	Poston-Springfield Brick Co., Springfield; upper 15 ft.	Trivoli shale overlying coal No. 8
238	Poston-Springfield Brick Co., Springfield; lower 20 ft.	" " " " " "
240	Purington Paving Brick Co., Galesburg; lower 20 ft.	Purington shale above coal No. 2
275	Alton Brick Co., Alton; lowermost shale	Purington shale overlying coal No. 2
270	Alton Brick Co., Edwardsville; entire shale	Trivoli shale overlying coal No. 8
279	Hydraulic Press Brick Co., Edgemont; lower shale	Shale underlying Collinsville limestone
268	Murphysboro Paving Brick Co., Murphysboro; lower shale	Shale 50 to 60 feet above Murphysboro coal
264	Albion Brick Co., Albion Plant No. 1; upper 20 ft.	Upper Pennsylvanian shale
269	Ford Brick and Tile Co., Harrisburg; entire shale	Shale above coal No. 6

with increasing amounts of clay minerals and grains of other minerals smaller than 0.002 mm. in diameter, i.e., fraction A. Shales with values that are higher or lower than the average for one green property usually have higher or lower values, as the case may be, for the other green properties.

The fact that all shales of equivalent fraction A content do not have the same green properties is evidence, if any is needed, that these properties are not controlled solely by the amount of clay mineral and by the amount of material finer than 0.002 mm. Shales with the same amount of fraction A but composed of different kinds of clay mineral will not have the same green properties. Shales, furthermore, with the same amount of fraction A and the same kind of clay mineral but with different textural attributes will not have the same green properties. Plotting of the fraction A content against ceramic properties permits an analysis of the effect of various factors on the ceramic properties of the shales.

Figure 4 shows that firing shrinkage increases with increasing amounts of fraction A. The relation of volume shrinkage to firing temperature for each shale is shown in figure 5. Cone values have been translated to degrees centigrade after Fairchild and Peters.[1]

The curves show that shales, e.g., Nos. 241, 278, and 280, which have large amounts of fraction A, tend to have a rapid sharp shrinkage at comparatively low temperatures. As the amount of fraction A decreases, shrinkage becomes more gradual. Shales with small amounts of fraction A attain maximum shrinkage at higher temperatures, and their shrinkage curves are less uniform than those with large amounts of fraction A. The curves were not plotted beyond the temperature of maximum shrinkage because complete data were not available. The limited data, however, indicate that shales rich in fraction A exhibit a longer interval between the temperature at which shrinkage is complete and that at which bloating becomes pronounced than do those with a small amount of fraction A.

Column (11) of table 2 shows that zero porosity is reached at successively higher cones in shales with decreasing amounts of fraction A. Shales with large quantities of fraction A show (fig. 6) a sharp, rapid loss of porosity at lower temperatures. As the content of fraction A decreases, porosity is lost more gradually and the minimum is reached at a higher temperature.

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[1] Fairchild, C. O., and Peters, M. F. Characteristics of Pyrometric Cones: Am. Ceramic Soc. Jour., vol. 9, no. 11, pp. 701–43, 1926.

The data in column (12), table 2, show no definite relationship between the amount of fraction A and the cone at which steel hardness is reached. Shales low in fraction A have the greatest temperature interval between cones of steel hardness and zero porosity and therefore would probably have the greatest firing range.

There is no correlation between fusion point and amount of fraction A. This is expected because the fusion point is closely related to the chemical composition of the clay minerals and may be influenced by small amounts of minor constituents.

Additional textural characteristics of the shales are given in table 3. Silicification (column (3)) refers to the presence of cryptocrystalline silica occurring around the edge of quartz grains and disseminated through the clay-mineral flakes which make up the shale. In the latter case the matrix, as seen by thin-section study, has a dense appearance so that it cannot be resolved into its component parts. Shales showing appreciable silicification (Nos. 237, 239, 227, 240, 275) in general have lower modulus of rupture and drying shrinkage than those without silicification but containing equal amounts of fraction A. The extremely fine silica acts as a cement binding together the particles composing fraction A, thereby having the effect of decreasing the proportion of fraction A. Particles small enough to be classed as fraction A are bound together into units large enough to act as components larger than fraction A, and, as a consequence, shrinkage and modulus of rupture are reduced.

Shales showing silicification could not

Table 2.—Ceramic Determinations

1	2	3	4	5	6	7	8	9	10	11	12	13	14
Sample No.*	Per cent fraction A	Water of plasticity in per cent	Modulus of rupture in lbs. per sq. in.	Modulus of rupture with 50 per cent sand in lbs. per sq. in.	Linear drying shrinkage in per cent	Volume drying shrinkage in per cent	Linear burning shrinkage in per cent†	Volume burning shrinkage in per cent†	Total linear shrinkage in per cent	Cone of zero porosity	Cone of steel hardness	P. C. E.	Color of fired product
278	100	31.0	583	215	8.8	28.8	12.0	31.8	20.8	1	08	10	red
280	95	30.8	483	227	7.1	22.9	12.0	31.8	19.1	1	08	7	red
229	95	31.8	477	284	7.6	24.5	11.9	31.7	19.5	1	08	14	red
241	95	38.5	1076	351	10.7	35.6	12.4	32.7	23.1	1	08	12	red
254	95	35.6	672	233	8.6	28.1	9.8	26.6	18.4	2	2	3	dark gray
237	95	29.5	291	169	6.3	20.0	12.6	33.3	18.9	1	08	8	red
239	90	28.8	302	183	5.9	18.8	12.4	32.7	18.3	1	08	10	red
285	85	26.3	439	220	5.8	18.5	11.0	29.6	16.8	1	08	7	red
262	80	28.2	465	223	6.8	22.0	11.5	30.8	18.3	2	08	14	red
281	80	28.4	424	225	7.1	22.7	11.6	30.9	18.7	3	08	7	red
227	75	26.4	236	100	4.5	14.2	11.9	31.6	16.4	3	02	8	red
242	70	27.1	290	147	6.2	19.8	10.9	29.3	17.1	3	08	11	red
231	65	24.3	302	221	5.5	17.3	11.8	32.3	17.3	3	08	15	red
213	65	28.5	269	152	5.5	17.6	11.4	30.4	16.9	3	02	9	red
243	60	28.0	398	220	5.8	18.4	11.3	30.2	17.1	3	06	13	red
238	60	26.6	260	181	5.0	15.8	10.9	29.3	15.9	3	03	9	red
240	60	26.2	216	156	4.6	14.5	11.9	31.6	16.5	3	03	10	red
275	50	23.5	188	92	4.4	13.9	10.2	27.6	14.6	6	02	11	red
270	50	24.0	214	133	4.8	15.0	9.7	26.5	14.5	5	03	9	red
279	45	23.0	424	219	5.5	17.4	6.5	18.3	12.0	5	08	10	red
268	40	21.3	218	170	4.7	14.9	7.7	21.3	12.4	7	2	14	dark gray
264	40	23.2	295	178	4.8	15.1	8.9	24.3	13.7	7	03	12	red
269	30	21.0	162	116	4.2	13.1	8.9	24.5	13.1	5	08	11	red

*The location of the sample is given in table 1.
†Values are those for maximum shrinkage.

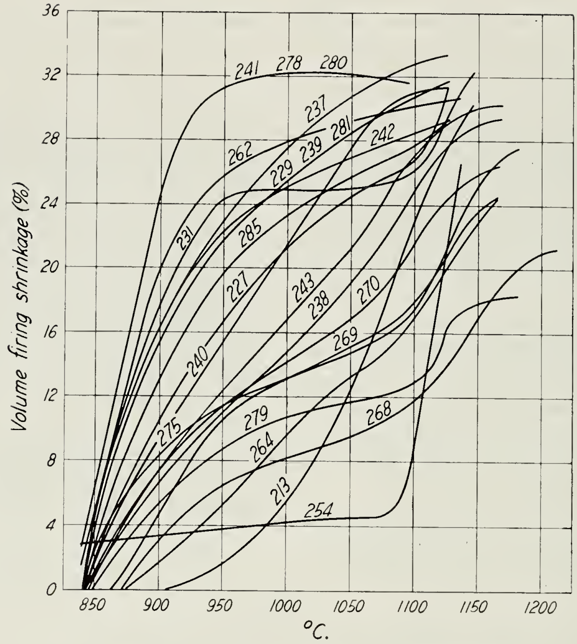

Fig. 5.—Volume firing shrinkage plotted against firing temperature.

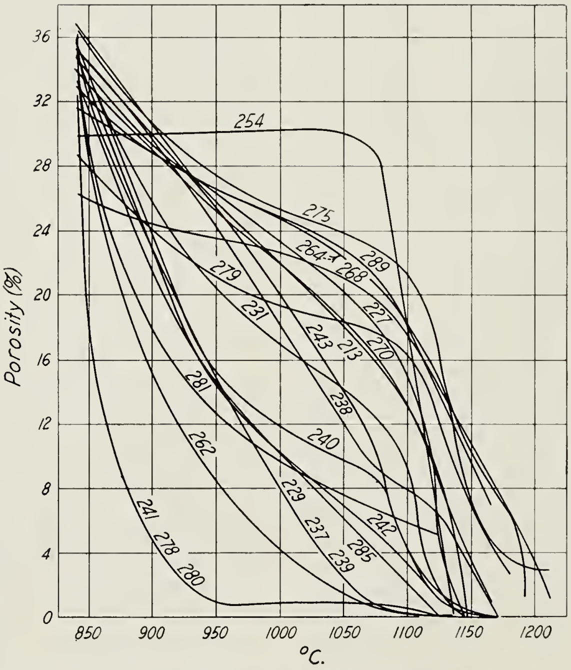

Fig. 6.—Porosity plotted against firing temperature.

Table 3.—Textural Characteristics

1	2	3	4	5	6	7	8	9
Sample No.	Per cent fraction A	Silicification*	Maximum size of clay minerals in mm.	Sorting of clay minerals†	Degree of uniformity of orientation of clay minerals in per cent	Maximum size of quartz grains in mm.	Sorting of quartz grains†	Quartz grains scattered (s) or in lenses (l)
278	100	L	.005	C	100			
280	95	L	.005	A	100	.03	C	s
229	95	L	.04	A	75	.03	C	s
241	95	L	.005	A	100	.03	C	s
254	95	L	.02	C	100	.03	C	s
237	95	M	.04	C	75	.03	C	s
239	90	M	.01	A	75	.03	C	s
285	85	L	.01	A	50	.04	C	s + l
262	80	L	.2	A	75	.04	C	s
281	80	L	.01	A	75	.12	A	s
227	75	M	.04	C	50	.06	C	s
242	70	L	.01	C	75	.1	C	l + s
231	65	L	.06	A	50	.05	C	s
213	65	L	.01	B	75	.03	C	s
243	60	L	.04	C	75	.06	C	s + l
238	60	M	.04	C	25	.06	C	s + l
240	60	M	.03	B	25	.12	C	s
275	50	M	.01	C	50	.12	C	s + l
270	50	L	.01	C	75	.1	C	l
279	45	L	.005	B	25	.06	C	s
268	40	L	.01	C	50	.1	C	l + s
264	40	L	.01	C	25	.2	C	l + s
269	30	L	.01	C	50	.06	C	s

* M = a large amount of silicification; L = a small amount of silicification.
†A = mainly near maximum size; B = mainly too small for size determination; C = unsorted.

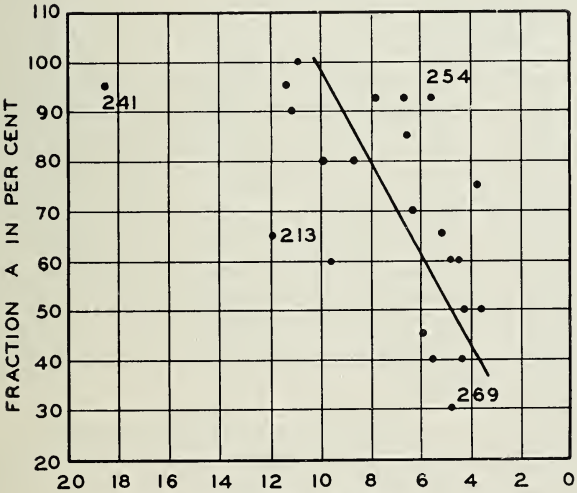

Fig. 7.—Base-exchange capacity plotted against amount of fraction A.

Table 4.—Base-Exchange Capacities*

Sample No.	Exchange capacity (m.e./100 gm.)	Sample No.	Exchange capacity (m.e./100 gm.)
278	10.9	231	5.1
280	7.7	213	11.9
229	11.3	243	9.5
241	18.5	238	4.8
254	5.5	240	4.4
237	6.5	275	3.5
239	11.1	270	4.2
285	6.4	279	5.9
262	9.9	268	5.4
281	8.7	264	4.3
227	3.7	269	4.6
242	6.2		

*Determinations made under the supervision of O. W. Rees, chemist, Illinois State Geological Survey.

readily be disaggregated and placed in suspension, and determinations of the amount of fraction A by sedimentation methods gave lower values than those determined by the study of thin sections.

There is no relation between the maximum size or uniformity of size of the clay-mineral particles (columns (4) and (5), table 3) in the raw shales and their ceramic properties. A consideration of silicification and a microscopic study of thin sections of shales in their natural state and after working with water (e.g., in a pug mill) but before firing provides a satisfactory reason for the absence of such a correlation. Shale No. 254, for example, is composed almost entirely of an unsorted assemblage of clay-mineral flakes with a maximum diameter of 0.02 mm. A thin-section study of the same shale after working but before firing shows it to be composed of clay-mineral flakes usually less than 0.002 mm. and with a maximum diameter of 0.007 mm. Working, therefore, has reduced the size of the clay-mineral flakes so that the size of particles in the raw shale and in the worked shale is not the same. Two shales containing clay-mineral particles of the same diameter, furthermore, would not exhibit the same ceramic properties if one were silicified because they would not break down to the same degree when worked in water. It is clear that any correlation of ceramic properties and particle size must be made on the basis of "effective particle size," that is, the size of the material after working. "Effective particle size" is not always the same as the particle-size make-up of the raw shale.

Degree of uniformity of orientation of clay minerals (column (6), table 3) refers to the uniformity of crystallographic orientation of the clay minerals in the shale. An analysis of this and other textural attributes listed in table 3 and not previously discussed shows no correlation with the ceramic properties listed in table 2. Other properties may be related to these textural attributes. The tendency of a shale, for example, to develop lamination planes during extrusion is probably controlled, at least in part, by the perfection of crystal form and degree of aggregate orientation of the clay-mineral flakes.

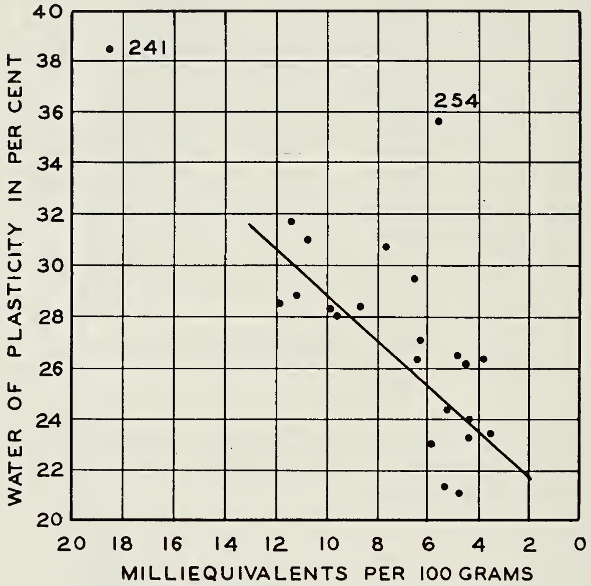

FIG. 8.—Water of plasticity plotted against base-exchange capacity.

MINERAL COMPOSITION

The mineral composition of the —0.002-mm. fraction of each shale was determined by X-ray and optical methods. Except for sample No. 241, the —0.002-mm. fraction of all shales is composed of the clay min-

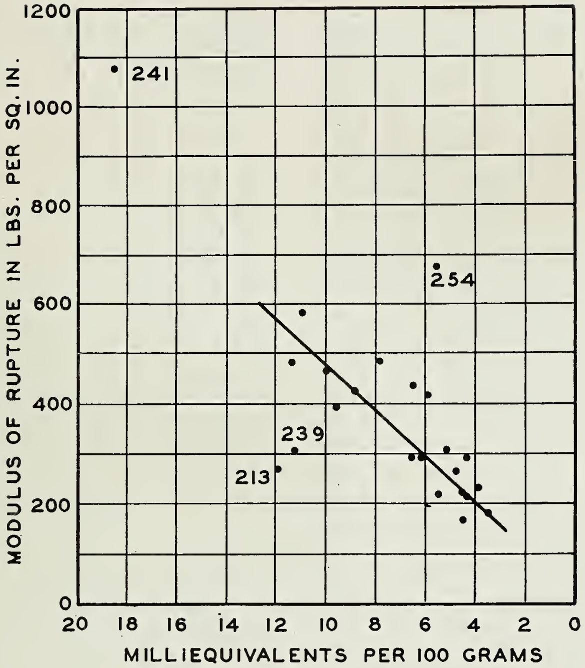

FIG. 9.—Modulus of rupture plotted against base-exchange capacity.

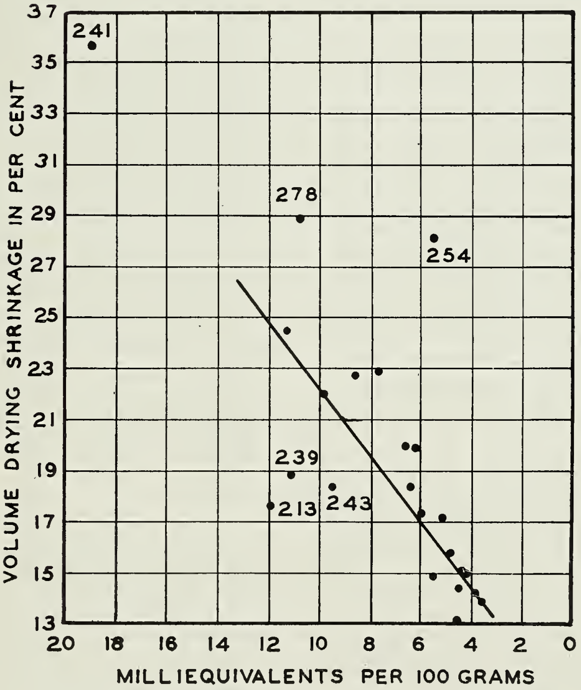

FIG. 10—Volume drying shrinkage plotted against base-exchange capacity.

erals, illite[2] and kaolinite, with minor amounts of chloritic mica, quartz, limonite, and organic material. Illite is more abundant than kaolinite. Shale No. 241 contains montmorillonite in addition to these constituents, and table 1 shows that this shale has much higher water of plasticity, modulus of rupture, and drying shrinkage than any of the other shales. This is in agreement with the conclusion of an earlier paper[3] that the presence of montmorillonite causes a clay to have high water of plasticity, bonding strength, and shrinkage.

Illite is known[4] to vary in its attributes, and before a specific correlation of properties and mineral composition is attempted for these shales, work in progress on the detailed properties of illite must be completed. Variations within the illite group of clay minerals must be known before a close correlation can be worked out.

The portion of the shales other than fraction A is made up essentially of quartz. A very few grains of orthoclase, pyrite, zircon, and tourmaline are also present. In none of the shales studied are the nonclay minerals, other than quartz, present in quantities large enough to affect the unfired properties.

BASE-EXCHANGE CAPACITY

The base-exchange capacity of each shale (table 4) was determined by the ammonium-acetate method. Figure 7 shows that, in general, the base-exchange capacity increases with the amount of fraction A. Because fraction A is approximately the component of the shale carrying the base-exchange capacity, that is, the clay minerals, this relationship is to be expected. Fraction A, however, also contains quartz and is a mixture of clay minerals with different exchange capacities so that a *close* relationship should not exist.

Montmorillonite possesses a higher base-exchange capacity than either illite or kaolinite. Shale No. 241, which contains montmorillonite, has a much higher exchange capacity than any other of the shales.

Figures 8, 9, and 10 show that, in general, the water of plasticity, modulus of rupture, and drying shrinkage increase with increasing base-exchange capacity. It follows, from a discussion similar to that already presented, that a rough correlation should exist between these ceramic properties and base-exchange capacity. A few shales, notably Nos. 254 and 213, possess exchange capacities which seem to be unrelated to their unfired properties. The reason for this absence of correlation is not yet known.

CONCLUSIONS

All of the shales of Pennsylvanian age in Illinois are composed principally of clay minerals (illite and minor amounts of kaolinite) and quartz. Chloritic mica, limonite, organic material, feldspar, and heavy minerals are minor and relatively unimportant components.

As far as mineral composition is concerned, the shales differ from each other chiefly in the relative abundance of the clay minerals and quartz. This gives a wide range in the ceramic properties of the Pennsylvanian shales. A close correlation exists between the amount of —0.002-mm. material plus all clay-mineral particles whatever their size, that is, fraction A and certain ceramic properties. As fraction A is largely the clay-mineral fraction, the relative abundance of clay minerals in the shale is an extremely important factor in determining ceramic properties.

One of the samples which contained montmorillonite in addition to illite and kaolinite exhibited higher values for unfired properties than any other sample studied.

Those shales which show silicification of the clay minerals have reduced values for their unfired properties. Certain other textural attributes, such as size and uniformity of orientation of clay-mineral particles, appear to have no influence on the ceramic properties considered herein.

Base-exchange capacity, which is a characteristic of the clay minerals, has a direct bearing on the unfired properties. The correlation, however, between base-exchange capacity and unfired properties cannot be close unless all materials have exactly the same clay-mineral composition and the same exchangeable base is present in each.

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[2] In an earlier paper, R. E. Grim, "Petrology of Pennsylvanian Shales and Noncalcareous Underclays, Associated with Illinois Coals, Including X-Ray Studies by P. F. Kerr and Chemical Studies by O. W. Rees, I-III:" Am. Ceramic Soc. Bull., vol. 14, no. 3, pp. 113–19; no. 4, pp. 129–34; no. 5, pp. 170–76, 1935, this component was designated as the sericite-like mineral.

[3] Grim, R. E., and Bray, R. H., Mineral Constitution of Various Ceramic Clays: Am. Ceramic Soc. Jour., vol. 19, no. 11, pp. 307–15, 1936.

[4] Grim, R. E., Relation of Composition to Properties of Clays: *ibid.*, vol. 22, no. 5, pp. 141–51, 1939.

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